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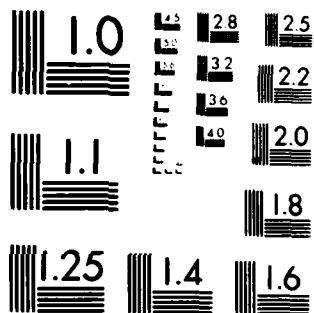
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REPORT DOCUMENTATION PAGE		READ INSTRUCTIONS BEFORE COMPLETING FORM
1. REPORT NUMBER Contract # N00014-75-C-0662	2. GOVT ACCESSION NO. NR # 031-715	3. RECIPIENT'S CATALOG NUMBER
4. TITLE (and Subtitle) "Mechanical Behavior of Particulate Composites"		5. TYPE OF REPORT & PERIOD COVERED FINAL REPORT
7. AUTHOR(s) Professor Oleg D. Sherby Dept. Materials Science & Engineering		6. PERFORMING ORG. REPORT NUMBER Stanford University
9. PERFORMING ORGANIZATION NAME AND ADDRESS Stanford University Stanford, California 94305 USA		8. CONTRACT OR GRANT NUMBER(s) N00014-75-C-0662 NR 031-715
11. CONTROLLING OFFICE NAME AND ADDRESS United States Office of Naval Research 800 N Quincy St., Arlington, VA. 22217		10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS
14. MONITORING AGENCY NAME & ADDRESS (if different from Controlling Office) United States Office of Naval Research 800 N Quincy Street, Arlington, VA 22217 Dr. Bruce A. MacDonald, Contract Monitor		12. REPORT DATE 1 August 1982
		13. NUMBER OF PAGES 26 pp
		15. SECURITY CLASS. (of this report) Unclassified
16. DISTRIBUTION STATEMENT (of this Report) "see distribution list - end of report"		15a. DECLASSIFICATION DOWNGRADING SCHEDULE
17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report)		
18. SUPPLEMENTARY NOTES		
19. KEY WORDS (Continue on reverse side if necessary and identify by block number) Powder metallurgy, particulate composites, mechanical properties, zinc, magnesium, cadmium, white cast iron, alumina, tungsten, iron carbide, superplasticity, phase transformation, thermal cycling, creep, plastic flow, ductility, modulus.		
20. ABSTRACT (Continue on reverse side if necessary and identify by block number) The program centers on a study of the mechanical properties of particulate metal-base composites. New processing techniques, principally through powder metallurgy processing, have been developed for attainment of ultrafine structures in particulate composites. Some of the discoveries made on the program are: 1) materials with high "threshold stresses" for creep ($\sigma_c = 10^{-3}$); 2) high specific modulus materials with isotropic properties ($E/\rho = 230 \times 10^6$ in); 3) superplastic white cast irons (>1000% elongation), and 4) enhanced densification of powders through phase transformation cycling.		

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FINAL REPORT

MECHANICAL BEHAVIOR OF PARTICULATE COMPOSITES

(N00014-75-C-0662, NR 031-715)

This is the Final Report to the Office of Naval Research program on "Mechanical Behavior of Particulate Composites", N00014-75-C-0662. The program was monitored by Dr. Bruce A. MacDonald. The principal investigator is pleased to acknowledge Dr. MacDonald's continuous guidance and encouragement throughout all phases of this program. His help, advice and constructive comments were always very much appreciated.

The Final Report is given in three sections. The first section summarizes the major findings of the program. The second section lists the publications, the presentations and patents; and the third section gives abstracts of each publication that have resulted from the program.

I. SUMMARY

The program centers on a study of the mechanical properties of particulate metal-base composites. A major emphasis is to learn how to optimize strength-ductility characteristics of such composites at low, intermediate and high temperatures. To this end, we have developed and continue to develop various processing techniques (principally through powder metallurgy processing) for attainment of ultrafine structures in particulate composites. This is because ultrafine structures in the form of fine particles of second phase and fine grain size, are generally highly desirable characteristics for enhancing mechanical properties. The choice

of second phase is an important factor in determining the mechanical properties of particulate composites. Not only is the size and distribution of second phase important but other properties also can be of paramount significance. Hardness, elastic properties, thermal expansion coefficient, and bonding characteristics of the second phase can significantly effect the properties of the final composite.

Our ultimate objective on this program was to develop new concepts and principles in a study of the mechanical properties of specially prepared particulate composites. Such principles may lead to the attainment of new and useful structural materials.

Two principal types of particulate composites have been emphasized in this research investigation. The first type of composite is based on the influence of a hard second phase, which remains hard at all temperatures, on the mechanical behavior of a given matrix material (e.g. Al_2O_3 particles in a zinc matrix, boron particles in a magnesium matrix). The second type of composite is based on the influence of a second phase, which is soft at high temperatures, on the mechanical behavior of a given matrix material (e.g. iron carbide in an iron matrix). In the following, we summarize our findings in these two composite systems and indicate the references (given in Section II) where further details on the subject are described.

1. Particulate Composites based on Presence of a Hard Second Phase

We cite the following examples of our work which may bear fruit in terms of possible technological use:



2.

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a) Composites with high structural strength and stability
at elevated temperature (Publications Nos. 1,2,4 and 5)

We have prepared, by a powder metallurgy-mechanical comminution (PMMC) process, large volume fraction particulate composites. When the second phase is hard (e.g. a zinc-alumina composite) such composites appear to exhibit a "threshold" creep stress. That is, such materials do not creep below a certain critical stress, a desirable characteristic for elevated temperature application. The threshold stress level is very high when the volume of second phase is large (e.g. $\sigma_0 \approx 10^{-3} E$, where E is Young's modulus).

b) Development of composites with high specific stiffness
(Patent No. 1)

Development of particulate composites, by our PMMC process, with high elastic stiffness properties. With magnesium as the base metal, we have prepared a Mg-B composite, containing 25 volume percent boron, with an elastic modulus of 11.4×10^6 psi. This value is about double that for most commercial magnesium alloys. This particular research has led to a patent issued to us in October, 1974. In order to ductilize the Mg-B composite we have added lithium as a solid solution addition to magnesium, and have successfully prepared Mg-Li-B particulate composites containing 10, 20 and 30 volume percent boron. The highest boron content Mg-Li composite exhibits a Young's modulus of 15.7×10^6 psi, and a very high specific stiffness of over 230×10^6 inches. A patent has been issued related to these elastically stiff light composites (U.S. Patent No. 3,955,933, issued 11 May 1976).

2. Particulate Composites Based on Presence of a Relatively Soft Second Phase (Fine Structure Superplasticity)

Considerable attention has been given by us to the properties and characteristics of iron base alloys containing a large amount of second phase in spherical form (cementite, Fe_3C). Such materials if processed correctly are superplastic at intermediate temperatures from the presence of fine ferrite grains. We summarize our contributions in this field which principally include studies of Fe-C alloys in the white cast iron composition range. In addition, this work has led us to write several reviews on fine structure superplasticity (Publications No. 8, 11, 12, 13, 14).

a) Refinement of microstructure in white cast irons
(Publications 7, 16, 17, 18)

New methods of developing ultrafine structures in white cast iron (2.4 and 3.0%C) are being pursued. Such studies may contribute to the development of improved high carbon tool steels. The first method of developing fine structures was by a hot and warm working procedure. The resulting microstructure was one of ultrafine ferrite grains with a microduplex structure of coarse and fine spheroidized cementite particles. Although these mechanically worked white cast irons exhibit superplastic properties at intermediate temperatures (600-700°C), cavitation is observed at tensile regions of coarse cementite particles. In order to eliminate cavitation, we have used rapid solidification rate (RSR) processing to develop uniformly fine cementite particles in white cast iron powders.

These powders have been warm pressed and subsequently worked at temperatures below the A_1 . This processing procedure results in a uniformly fine structure of cementite in a fine ferrite grain matrix. Mechanical tests reveal improved superplastic properties as well as improved room temperature properties.

b) Enhancement of densification of powders by thermal cycling (Publication 15, Patent 2)

A very significant result obtained during the last year of the program was the demonstration that densification of powders (and solid state bonding) can be enhanced through multiple phase transformations. A patent disclosure has been submitted to Stanford University based on our new (O.N.R. supported) findings.

The above discovery was based principally on experiments with rapidly solidified white cast iron powders. This new work demonstrates that ferrous powders can be readily densified at low temperatures and short times at low externally applied stresses, under thermal cycling conditions. The end-results are dense compacts that still retain fine structures with highly useful mechanical properties. This new knowledge can be applied to the case of highly-alloyed tool steels where fine carbide sizes and fine grain sizes are known to be highly beneficial to enhancing room temperature properties (e.g. wear resistance and fatigue resistance). Such fine structures are inherent in powders that are rapidly solidified. Utilizing the enhanced densification offered by multiple transformation plasticity, consolidation at temperatures where

little carbide or grain coarsening occurs is possible. The low stresses needed for such densification allows for ready application in current HIP equipment. The principle of enhanced densification through transformation plasticity can also be applied to solid state welding of ferrous materials at low temperatures.

- c) Development of particulate composites which can be selectively heat treated so that only one of the constituents (the minor one) transforms
(Publications 3 and 19)

We have illustrated the feasibility of obtaining dense compacts of iron and white cast iron by warm pressing powders of these constituents below the critical temperature. An important finding was that this high densification is enhanced by the presence of a fine structure in one of the powders (white cast iron) (Publication No. 19). Selective heat treatment is accomplished by heating the compact just above the A_1 , transforming only the white cast iron regions, followed by quenching. The result is a composite consisting of high carbon martensite in a matrix of untransformed low carbon ferrite. High strengths and high hardness, with good ductility, composites can be attained in this manner. The concept of solid state bonding dissimilar ferrous materials, followed by selective heat treatment, can be readily applied to laminate composites and is the subject of one of our publications (Publication No. 3).

d) Superplastic forming of particulate composites

Because particulate composites can have a threshold creep stress at elevated temperature (i.e. they are strain rate insensitive), they fail with little plasticity in tension leading to poor formability qualities. Our theoretical modelling of internal stress-assisted plastic flow would suggest that particulate composites may be superplastic when deformed under thermal cycling conditions. The internal stress arises from the difference in thermal expansion coefficient of particle and matrix phases. This concept is currently being tested experimentally. If verified, a novel concept of shaping particulate composites into unusual and complex shapes may be possible.

II. PUBLICATIONS, PRESENTATIONS AND PATENTS

A. PUBLICATIONS

1. "Yield Points and Strain Aging in Hexagonal-Based Particulate Composites", I. C. Huseby, S. E. Hsu, T. R. McNelley, G. R. Edwards, D. Francois, J. C. Shyne and O. D. Sherby, Metall. Trans. A, 6A, 1975, 2005-2008.
2. "Diffusion Controlled Deformation of Particulate Composites", Glen R. Edwards, Terry R. McNelley and Oleg D. Sherby, Phil. Mag., 32, 1975, 1245.
3. "Superplastic Hot Pressing of White Cast Iron", R. D. Caligiuri, R. T. Whalen and O. D. Sherby. The Int'l. J. of Powder Metall., 12, 9 (January, 1976).
4. "A Microstructural Correlation Between the Mechanical Behavior of Large Volume Fraction Particulate Composites at Low and Elevated Temperature", T. R. McNelley, G. R. Edwards and O. D. Sherby, Acta Met., 25, 1977, 117-124.
5. "Mechanical Behavior of Cadmium-Boron and Cadmium-Tungsten Particulate Composites", S. E. Hsu, G. R. Edwards, J. C. Shyne and O. D. Sherby, J. Mat. Sci., 12, 1977, 131-140.
6. "Superplastic White Cast Irons", J. Wadsworth and O. D. Sherby, Foundry Manufacturing and Technology, 106 (10), October, 1978, 59-73.
7. "The Development of Ultrafine, Superplastic Structures in White Cast Iron", J. Wadsworth, L. E. Eiselstein and O. D. Sherby, Mat. Eng. Appl., 1, 1979, 143-153.
8. "Damascus Steels Rediscovered?", O. D. Sherby, Trans. of the Iron and Steel Institute of Japan, 19, 1979, 381-390.
9. "Superplastic Bonding of Ferrous Laminates", O. D. Sherby, J. Wadsworth, R. D. Caligiuri, L. E. Eiselstein, B. C. Snyder and R. Whalen, Scripta Met., 13, 1979, 941-946.

10. "Superplasticity - Prerequisites and Phenomenology", J. Wadsworth, T. Oyama and O. D. Sherby, Proceedings of the Sixth Inter-American Conference on Materials Technology, Volume 2, American Society for Mechanical Engineers, New York City, N.Y., 1980.
11. "Superplastic Materials - Their Preparation and Their Application", The Materials Policy, Research and Development Act of 1979, Ninety-Sixth Congress, First Session on H.R. 2743, June 25, 26, 28, 1979, pp 55-72, U.S. Government Printing Office, Washington, 1979 (No. 65).
12. "Fundamentals of Superplasticity and its Application" (by Oleg D. Sherby, R. D. Caligiuri, E. S. Kayali and R. A. White, in Advances in Metal Processing, Proceedings of the Twenty-Fifth Sagamore Conference, Plenum Press, 1981, 133-171.
13. "Ultrahigh Carbon Steels - Their Properties and Potential", Oleg D. Sherby, Bruce A. MacDonald and Edward C. Van Reuth, Naval Research Reviews, 33, 1981, 48-61.
14. "On the Bulat - Damascus Steels Revisited", Jeffrey Wadsworth and Oleg D. Sherby, Prog. in Mat. Sci., 25, 1981, 35-68.
15. "Enhanced Densification of White Cast Iron Powders by Cyclic Phase Transformations under Stress", Oscar A. Ruano, Jeffrey Wadsworth and Oleg D. Sherby, Metall. Trans., 13A, 1982, 355.
16. "Superplasticity in Rapidly Solidified White Cast Irons", Oscar A. Ruano, Lawrence E. Eiselstein and Oleg D. Sherby, Metall. Trans. A, 13A, 1982, 1785.
17. "Structural Characterization of Rapidly Solidified White Cast Iron Powders", L. E. Eiselstein, O. A. Ruano and O. D. Sherby, J. Mat. Sci., 1982, in press.
18. "Strength and Ductility at Room Temperature of Rapidly Solidified White Cast Irons", Oscar A. Ruano, Lawrence E. Eiselstein and Oleg D. Sherby, accepted for publication in Powder Metallurgy.

19. "Microstructure and Mechanical Properties of Rapidly Solidified White Cast Iron Powder and Iron / White Cast Iron Particulate Composites", Lawrence E. Eiselstein, Ph.D. Thesis, December, 1982.

B. PRESENTATIONS

1. Paper on "Creep Mechanisms in Cadmium", by G. Edwards, S. Hsu and O. D. Sherby, presented at AIME Annual Meeting, Las Vegas, Nevada, February 25, 1976. (Abstract in J. of Metals, December, 1975).
2. Paper on "A Correlation Between Microstructure and Mechanical Behavior of Zinc-Based Particulate Composite Materials", T. R. McNelley, G. R. Edwards and O. D. Sherby, presented at AIME Annual Meeting, Las Vegas, Nevada, February 24, 1976. (Abstract in J. of Metals, December, 1975).
3. Paper presented on "Structure and Mechanical Properties in Ferrous Composites", by Lawrence Eiselstein and Oleg D. Sherby, Western Metals Congress, Los Angeles, March 16, 1977.
4. "Mechanical Working of Cast Iron", by J. Wadsworth and O. D. Sherby, paper presented at WESTEC 1978, March 21, 1978, Los Angeles, California.
5. Talk presented at the Annual Meeting of the AIME, New Orleans, LA, February 20, 1979, on "Pressure Sintering Kinetics of Iron Powders and Superplastic Ultrahigh Carbon Steel Powders", R. D. Caligiuri and O. D. Sherby.
6. "Development of Superplastic Microstructure and Microstructure-Property Relations", talk by Oleg D. Sherby at 109th Annual Meeting of AIME, Las Vegas, February 26, 1980.
7. "Superplasticity-Principles and Prerequisites", talk presented at National Bureau of Standards, Gaithersburg, Maryland, March 28, 1980, by Oleg D. Sherby.

8. "Superplasticity - Prerequisites and Phenomenology",
Keynote lecture by Oleg D. Sherby at Century 2 - Emerging
Technology Conferences, American Society for Mechanical
Engineers, August 12-15, 1980, San Francisco.
9. Talks given by Oleg D. Sherby on "Superplasticity: Prerequisites,
Phenomenology and Applications", to Westinghouse Research
Laboratory, Monroeville, Pennsylvania (October 29, 1980), to
NATO Structural Materials Conference, Bremen, Germany
(April 9, 1981), to General Motors Research Center, Warren,
Michigan (June 10, 1981) and to International Harvester
Corporate Research Center, Chicago, Illinois (July 15, 1981).
10. Talk given by Oleg D. Sherby "Synthesis and Characterization
of Superplastic Alloys", one of keynote talks at International
Symposium on Superplastic Forming of Structural Alloys, San
Diego, California (June, 1982); also similar talk given in
Tokyo, Japan and Taipei, Taiwan in April-May, 1982.

C. PATENTS

1. "Magnesium-Boron Particulate Composites", by Oleg D. Sherby,
Irvin Huseby, Robert T. Whalen and Steven Robinson, United States
Patent #3,955,933, dated May 11, 1976.
2. "Method of Consolidation of Iron-Based Alloy Powder by Cyclic
Phase Transformation under Pressure", Stanford Docket S80-57,
Office of Technology Licensing, Stanford University, submitted to
United States Patent Office in March, 1982.

III.

A B S T R A C T S
O F
P U B L I C A T I O N S
R E S U L T I N G
F R O M
T H E P R O G R A M

1. Yield Points and Strain Aging in Hexagonal-Based Particulate Composites

I. C. HUSEBY, S. E. HSU, T. R. McNELLEY, G. R. EDWARDS, D. FRANCOIS,
J. C. SHYNE, AND O. D. SHERBY

Initial and strain-aging yield points have been observed and studied for three hexagonal-based particulate composites: Cd-30 vol pct B, Mg-25 vol pct B, and Zn-15 vol pct Al_2O_3 . The strain-aging yield points begin to appear at 0.5 to $0.6T_m$ and persist up to the melting temperature. These effects are associated with the large volume fraction of hard particles, which are considered to be a primary source of dislocations and vacancies; strain aging yield points are attributed to the resulting decrease in mobile dislocation density, by annihilation or interaction with imperfections other than impurity atoms, which occurs during aging after straining.

Reprinted from
THE PHILOSOPHICAL MAGAZINE, Vol. 32, No. 6, p. 1245, December 1975

2. Diffusion-controlled deformation of particulate composites

By G. R. EDWARDS

Department of Mechanical Engineering, Naval Postgraduate School,
Monterey, California, U.S.A.

T. R. McNELLEY

Department of Mechanical Engineering, University of Wyoming,
Laramie, Wyoming, U.S.A.

and O. D. SHERBY

Department of Materials Science and Engineering, Stanford University,
Stanford, California, U.S.A.

[Received 13 June 1975 and after revision 17 September 1975]

ABSTRACT

Unusual mechanical effects at elevated temperatures have been observed in a composite system of zinc containing large volume fractions (0.05 to 0.30) of small ($<1 \mu m$) Al_2O_3 or W particles. These materials are dispersion-weakened in the temperature range 0.3 to $0.7T_m$, and show marked insensitivity of flow stress to changes in strain rate or temperature at about $0.7T_m$. Strain-ageing yield points are also observed at $0.6T_m$. An explanation for such behaviour proposes that, above a threshold stress, mobile dislocations are generated at particle-matrix interfaces by local incompatibility stresses, and that these dislocations have a dominant effect on the nominal flow stress in the region 0.3 to $0.7T_m$. A constitutive equation reminiscent of the Ansell-Wcertainan theory for creep of dispersion-strengthened materials is derived from the model, and compared with experimental creep data.

Superplastic Hot Pressing of White Cast Iron

R. D. CALIGIURI,* R. T. WHALEN,** and O. D. SHERBY***

Int'l. J. Powder & Powder Tech., 12, No.1, January, 1976

ABSTRACT

A new method of sintering high carbon ferrous powders using hot pressing techniques is described. The process is called "superplastic hot pressing". This technique utilizes the exceptional weakness of superplastic structures, and permits the production of dense compacts at low temperatures and pressures. White cast iron powders (2.6%C) are hot pressed in air for 2-5 hours at 69-193 MPa (10,000-28,000 psi) and 600-700°C (1112-1292°F) into compacts 90 to 99% dense. The mechanical properties of this material are shown to be commensurate with as-cast white cast iron (a fracture strength at room temperature of 1241 MPa (180,000 psi)). Strain rate change and stress relaxation tests on hot pressed white cast iron at 650°C revealed it to be highly strain rate sensitive ($m = 0.3$ in $\sigma = K\dot{\epsilon}^m$) indicating that sintering by superplastic type flow mechanisms occurred. It is predicted that a finer structure than that developed in our cast iron powders would lead to $m = 0.5$ (ideal superplastic state) and therefore would result in ideal superplastic hot pressing.

4.

A MICROSTRUCTURAL CORRELATION BETWEEN THE MECHANICAL BEHAVIOR OF LARGE VOLUME FRACTION PARTICULATE COMPOSITES AT LOW AND HIGH TEMPERATURES

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and

O. D. SHERBY

Department of Materials Science and Engineering, Stanford University,
Stanford, CA, U.S.A.

Abstract—Unusually high yield strengths, up to $E/110$ where E is Young's modulus, and high rates of work hardening have been observed in zinc-based particulate composites at low homologous temperature ($T \approx 0.1 T_M$). These composites contain a large volume fraction (~ 0.3) of uniformly distributed W particles ($< 0.6 \mu\text{m}$ in dia.) or Al_2O_3 particles (0.6 or $0.3 \mu\text{m}$ in dia.) in a fine grained zinc matrix. At low homologous temperatures, particles are assumed to be sources of geometrically necessary dislocations, and work hardening due to these dislocations is evaluated on the basis of a single parameter, the geometric slip distance λ_G . A model previously developed by the authors for elevated temperature (up to $0.8 T_M$) deformation in these composites also considers the particles to be dislocation sources in a fine, stable microstructure. The geometric slip distance λ_G is incorporated into the model for elevated temperature deformation and the mechanical behavior of these particulate composites is then consistently and accurately described for temperatures from 0.1 to $0.8 T_M$.

JOURNAL OF MATERIALS SCIENCE 12 (1977) 131-140

5.

Mechanical behaviour of cadmium-boron and cadmium-tungsten particulate composites

SHU-EN HSU

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J. C. SHYNE, O. D. SHERBY

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California, USA

Mechanical behaviour of particulate composites of cadmium containing 0.6 to $3 \mu\text{m}$ size particles of boron and tungsten (up to $30 \text{ vol } \%$) were studied from -196 to 260°C (0.13 to $0.9 T_m$). The marked strengthening of cadmium by the presence of fine particles is attributed to significant grain size and texture strengthening effects as well as to dispersion hardening effects.

6.

Superplastic White Cast Irons

That cast iron cannot be worked has been accepted as an immutable fact. Two Stanford University scientists recently proved, however, that we should change our minds, for white iron can be formed and can become superplastic. This article describes some of the remarkable work — a significant breakthrough.

By J. WADSWORTH / Research Associate, and
O. D. SHERBY, Professor, Dept. of Materials Science and Engineering,
Stanford University, Stanford, Calif.

7. The development of ultrafine, superplastic structures in white cast irons

Materials in Eng. Applications, 1, 1979, 143

J. WADSWORTH, L. E. EISELSTEIN and O. D. SHERBY†

At room temperature white cast irons are normally regarded as very brittle materials due to a high content of brittle iron carbide. However, it has long been known that at high temperatures white cast irons and iron carbide are deformable by various hot working processes. Recent work at Stanford has demonstrated that a series of thermomechanical treatments, including extensive warm working, refines the as-cast structure so as to impart unusual and highly beneficial properties to the cast irons. In particular the worked white cast irons are considered to be superplastic and there is a great improvement in room temperature properties.

This processing procedure may, therefore, have significant technological applications. Though the material is in an early development stage the paper suggests that the well-established properties of old materials can be drastically changed by modifying their microstructure.

Damascus Steel Rediscovered?*

Trans. Iron & Steel Inst., Japan
19, 1979, 381-390

By Oleg D. SHERBY**

Synopsis

The outstanding characteristics of Damascus steel are legend. Not only beautiful in design, they were known for their strength, toughness and retention of their cutting edge. The forgotten art of making these steels may have recently been rediscovered at Stanford University. The metallurgical key to the development of such steels is the use of ultrahigh carbon contents and the use of thermal-mechanical processing procedures for attainment of sub-micron microstructures. Such steels are superformable (superplastic) at warm temperature and strong and ductile at room temperature. The preparation and properties of fine-grained ultrahigh carbon steels will be described, including their use in making laminated composites similar to the structures noted in Japanese laminated tools and welded Damascus steel.

Scripta METALLURGICA Vol. 13, pp. 941-946, 1979
Printed in the U.S.A.

Pergamon Press Ltd.
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SUPERPLASTIC BONDING OF FERROUS LAMINATES

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(Received July 9, 1979)

(Revised August 24, 1979)

Introduction

It is the purpose of this paper to demonstrate that both similar and dissimilar ferrous materials may be bonded below the eutectoid (A_1) temperature (727°C) and to demonstrate the advantages of bonding at such low temperatures. Special emphasis will be placed on the role of superplastic structures in enhancing bonding. Of special interest to the work described herein are steels designated as ultrahigh carbon (UHC) steels. These steels contain between 1 and 2.1% C. It has been shown that these steels, after appropriate thermomechanical processing, can be readily made superplastic (1-5). These steels, having a structure of fine (~ 0.1 - $0.5\mu\text{m}$ diameter) spheroidized cementite particles in a fine-grained (~ 1 - $2\mu\text{m}$) ferrite matrix, are not only superplastic but also exhibit good room temperature properties (3-5). In addition, such steels can be heat treated to very high hardnesses by quenching from just above the A_1 temperature (6).

Previous attempts to solid-state diffusion bond dissimilar ferrous materials have invariably been carried out above rather than below the A_1 temperature (7-12). Attempts to solid-state diffusion bond similar ferrous materials below the A_1 are also limited although Caligiuri et al. (13) have demonstrated the feasibility of isostatically pressing white cast iron powders (made fine-grained by liquid atomization) below the A_1 temperature. Bonding below, rather than above, the A_1 temperature has a number of significant advantages. First, it may be undesirable to exceed the A_1 temperature in one of the ferrous materials being bonded. This is because certain properties can be permanently lost if such a material is subjected to temperatures above the A_1 temperature (e.g. texture, superplasticity in eutectoid steels, fine microstructures, etc.). Second, if bonding is carried out below the A_1 transformation temperature, then selective heat treatment of the resulting composite is possible if appropriate starting materials are chosen. An example would be the bonding of an ultrahigh carbon steel to interstitial-free iron. At temperatures below the A_1 , minimal diffusion of carbon will occur across the interface of these two materials. This is because nearly all the carbon is at all times combined as cementite in the UHC steel. If such a composite is then heated to just above the A_1 transformation temperature, and quenched, the interstitial-free iron will be unaffected, but the ultrahigh carbon steel will be hardened by transformation to martensite plus cementite. This concept of selective heat treatment can be utilized to produce dual-hardness composites.

It is of interest that considerable effort has been directed to the diffusion bonding of the superplastic Ti-6Al-4V (14) alloy and indeed to the simultaneous superplastic forming and diffusion bonding (SPF/DB) of this alloy (15). We will demonstrate in this paper that this potential also exists in the iron-carbon system.

10.

SUPERPLASTICITY: PREREQUISITES AND PHENOMENOLOGY

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ABSTRACT

The term superplasticity has been used to describe extraordinary elongations (several hundred percent) obtained during tensile deformation of polycrystalline materials. Mostly a scientific curiosity fifteen years ago, fine-structure superplastic materials are now being used in a number of industrial applications including near-net shape forming and solid-state bonding. The various structural prerequisites for fine-structure superplasticity are evolving rapidly and the phenomenology of superplastic flow is well documented. This knowledge, coupled with advances made in understanding normal plastic flow in crystalline solids (diffusion-controlled dislocation creep), permits the prediction of methods for enhancing and optimizing superplasticity in materials.

Statement of

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11.

SUPERPLASTIC MATERIALS - THEIR PREPARATION
AND THEIR APPLICATION

presented to

House Committee on Science and Technology

as part of a symposium on

"Materials of the Future - Their Impact on Our Society"

June 25, 1979

IN: ADVANCES IN METAL PROCESSING

Proceedings of the 25th Sagamore Conference,
Plenum Press, 1981, 133

12.

FUNDAMENTALS OF SUPERPLASTICITY AND ITS APPLICATION

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Abstract

The term superplasticity has been used to describe extraordinary elongations (several hundred percent) obtained during tensile deformation of polycrystalline materials. Mostly a scientific curiosity fifteen years ago, superplastic materials are now being used in a number of industrial applications, including near-net-shape forming and solid state welding. Two types of superplastic flow have been observed: internal stress superplasticity and fine structure superplasticity. Both rely on one common characteristic: a high sensitivity of the flow stress to the strain rate. This is a necessary but not sufficient condition for superplasticity. The various structural prerequisites for fine structure superplasticity are evolving rapidly and the phenomenology of superplastic flow is well documented. This knowledge, coupled with advances made in understanding normal plastic flow in crystalline solids (diffusion controlled dislocation creep), permits predicting methods for enhancing and optimizing superplasticity in materials.

13.

Ultrahigh Carbon Steels— Their Properties and Potential

by

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The preparation, properties and potential of ultrahigh carbon steels are described in the following article. It appears that the metallurgical key to the development of fine-grained, ultrahigh carbon steels is the use of thermal mechanical processing for attainment of submicron microstructures. Such steels behave in a superplastic manner at warm tempera-

tures and are strong and ductile at room temperature. Their similarity to the ancient Damascus steels and Japanese laminated tools is noted with the recognition that the forgotten art of making the Damascus steels may have been rediscovered in this program at Stanford University.

14.

ON THE BULAT—DAMASCUS STEELS REVISITED†

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(Submitted 2 June 1980)

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"... Our warriors will soon be armed with bulat blades, our agricultural labourers will till the soil with bulat plough shares, our artisans will use tools fashioned of bulat and bulat will supersede all steel now employed for the manufacture of articles of special sharpness and endurance."

Anosoff, 1841, Russia

"... Unable to complete these researches, the author nevertheless hopes that his paper may again arouse in this country an interest in damascene steel and that a further step towards the fulfilment of Anosoff's forecast may thus be achieved."

Belaiew, 1918, England

PREFACE

In 1975 at Stanford University it was discovered that ultrahigh carbon (UHC) steels, that is, steels containing between 1–2.1% C, could be made both superformable (i.e. superplastic) at warm temperatures and strong and ductile at room temperature.⁽²⁾ At a later date, it came to the authors' notice that these steels were in fact similar, especially in carbon content, to Bulat or Damascus steels,[§] and this prompted our interest in the history and metallurgy of such steels.

Metall. Trans. A, 13A, 1982, 355

15. Enhanced Densification of White Cast Iron Powders by Cyclic Phase Transformations under Stress

OSCAR A. RUANO, JEFFREY WADSWORTH, and OLEG D. SHERBY

It is shown that densification of white cast iron powders under stress can be enhanced by multiple phase transformations through thermal cycling. This enhancement occurs by accelerated creep flow during phase changes (transformation superplasticity). The approximate stress range where transformation-assisted densification can occur is shown to be between 1.7 MPa (250 psi) and 34.5 MPa (5000 psi). Below 1.7 MPa insufficient strain occurs during phase transformation to cause significant densification even after many transformation cycles. Above 34.5 MPa, densification occurs principally by normal slip creep. Transformation warm pressing of white cast iron powders leads to dense compacts at low pressures and short times. In addition, because the transformation temperature is low, the ultrafine structures existing in the original powders are retained in the densified compacts.

Superplasticity in Rapidly Solidified White Cast Irons

OSCAR A. RUANO, LAWRENCE E. EISELSTEIN, and OLEG D. SHERBY

Superplastic properties of three different composition white cast irons were investigated in the temperature range of 630 to 725 °C. Fine structures consisting of 1 to 2 μm ferrite grains were developed in these materials by consolidation of rapidly solidified powders at intermediate temperatures below the A_1 critical temperature. Tensile elongations of 1410 pct were found for a 3.0 pct C + 1.5 pct Cr white cast iron, 940 pct for a 3.0 pct C white cast iron, and 480 pct for a 2.4 pct C white cast iron when tested at 700 °C and at a strain rate of 1 pct per minute. The superplastic white cast irons exhibited a high strain rate sensitivity exponent, m , of 0.5 and activation energies for plastic flow were found to be nearly equal to the activation energy for grain boundary self-diffusion in iron. These observations are in agreement with the creep behavior of superplastic materials controlled by grain boundary diffusion.

17.

STRUCTURAL CHARACTERIZATION OF RAPIDLY SOLIDIFIED WHITE
CAST IRON POWDERS

by

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ABSTRACT

Three white cast iron alloy powders (2.4%C, 3.0%C, and 3.0%C+1.5%Cr) manufactured by a rapid solidification processing technique were investigated. It was found that the microstructures of all three alloy powders were similar. The primary constituent of these powders was found to be retained austenite. Only small amounts of carbide and martensite were found in the rapidly solidified white cast iron powders. The primary austenite cells and dendrites that nucleate and grow from the melt are retained upon cooling to room temperature with little carbide precipitation. The low volume fraction of martensite found was due to the high carbon concentration of the austenite. A fine dispersion of carbides in an austenite matrix is formed as a result of the solidification of the eutectic liquid in the intercellular and interdendritic regions. The relative proportion of primary austenite to eutectic can be explained by the carbon segregation that occurs during the solidification of the primary austenite. Annealing of the powders at 650°C transforms the metastable austenite into alpha iron and carbide. The carbides have a bimodal distribution with small carbides precipitating within the primary austenite cells and dendrites and large carbides precipitating within the intercellular and interdendritic regions.

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18. STRENGTH AND DUCTILITY, AT ROOM TEMPERATURE, OF RAPIDLY
SOLIDIFIED WHITE CAST IRONS

by

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ABSTRACT

The room temperature mechanical properties of three white cast iron powder compacts (2.4%C, 3.0%C and 3.0%C+1.5%Cr) were investigated. The compacts, obtained by warm compaction and rolling of rapidly solidified powders, were found to have a fine microstructure which strongly influenced the mechanical properties. Large strain to failure values in compression for the as-pressed compacts were obtained in the order $\epsilon = 0.25$. Tensile ductility of over 7% was obtained in rapidly solidified 3.0%C white cast iron which contrasts with nil ductility for the as-cast material. It is shown that alloying and heat treatment influences the yield strength and ductility of the white cast irons.

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